

DECLARATION

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Saitama-shi, Saitama, Japan, certify that I know well
both the English and Japanese languages and that the
attached English language translation is a true and
correct translation of Japanese Patent Application No.
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knowledge and belief.

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【TYPE OF DOCUMENT】 SPECIFICATION

【TITLE OF THE INVENTION】

WATER BASE PRIMER AND METHOD FOR BONDING

【SCOPE OF THE CLAIM(S)】

5 【Claim 1】

A water base primer, characterized by comprising a polychloroprene and an EVA resin as essential components.

 【Claim 2】

 The water base primer according to Claim 1, wherein
10 mass ratio of ethylene in the EVA resin is at least 60%.

 【Claim 3】

 The water base primer according to Claim 1 or 2,
wherein proportion of solid content is adjusted within a
range of from 10 to 30 wt%.

15 【Claim 4】

 The water base primer according to any one of Claims
1 to 3, which further comprises an adhesive resin
emulsion.

 【Claim 5】

20 A method for bonding an adherend, which is
characterized by using the water base primer as defined
in any one of Claim 1 to 4.

【DETAILED DESCRIPTION OF THE INVENTION】

 【Technical Field to which the Invention Belongs】

25 The present invention relates to a novel water base
primer which is applied on a surface of an adherend

before an adhesive is applied in order to obtain enough adhesive strength, when the adherend such as an organic material or a porous organic material is bonded, especially when bonding is carried out by means of a polychloroprene type water base adhesive. The present invention also relates to an adhesion place for bonding the organic material, the porous organic material or the like which is a material of parts such as a shoe, a sport apparatus, packing, automobile, building material, etc., by means of this water base primer.

[Prior Art]

Heretofore, as a method for bonding an adherend which is hard to adhere such as an organic material or a porous organic material, an adhesion method wherein a surface of adherend is coated with a solvent type primer, followed by employing a solvent type adhesive such as a polychloroprene, has been mainly employed. However, the solvent type adhesive contains an organic solvent such as toluene, ethyl acetate or methyl ethyl ketone, whereby it is undesirable for safety and health of workers and for the environment. Therefore, demand for a water base adhesive and a water base primer is increasing year by year. Some water base adhesives have been proposed (for example, Patent Documents 1 and 2). However, with respect to the primer, one which can be used practically has not been known. In actual, a solvent type primer is used (for example Patent Document 3).

【Patent Document 1】JP-A-2001-64616

【Patent Document 2】JP-A-2001-26756

【Patent Document 3】JP-A-2001-164176

【Problems that the Invention is to Solve】

5 Under such present circumstances, it is an object of the present invention to provide a water base primer having adhesive strength comparable with the conventional solvent type adhesive.

【Means of Solving the Problems】

10 The present inventors have conducted studies to attain the above object, and as a result, have found that a composition comprising a polychloroprene and an EVA resin as main components, has excellent adhesive strength as the water base primer. Thereby, the present invention
15 has been accomplished.

Namely, the present invention provides a water base primer characterized by comprising a polychloroprene and an EVA resin as main components and a method for bonding an adherend by means of it.

20 Now, the present invention will be described in further detail. The polychloroprene in the present invention means a single monomer of 2-chloro-1,3-butadiene(hereinafter referred to as chloroprene) or a copolymer of the chloroprene with a monomer which is
25 copolymerizable with the chloroprene. The copolymer here includes a graft copolymer obtained by graft polymerization of another polymer in the presence of the

polychloroprene.

As the monomer copolymerizable with chloroprene, for example, 2,3-dichloro-1,3-butadiene, 1-chloro-1,3-butadiene, butadiene, isoprene, styrene, acrylonitrile, 5 acrylic acid and its ester or methacrylic acid and its ester may be mentioned. If necessary, two or more of them may be employed in combination.

A method for polymerizing the polychloroprene is not particularly restricted. However, a method of emulsion 10 polymerization is common and simple.

In order to accomplish the present invention, it is necessary to disperse the polychloroprene in water. However, in a case of emulsion polymerization, the obtained polychloroprene latex can be used as it is.

15 An emulsifying agent and/or a dispersing agent which is used for emulsion polymerization of the polychloroprene latex is not particularly restricted. Various types of anionic type, nonionic type or cationic type which is usually used for the chloroprene latex may 20 be used. As the anionic type, there is, for example, a carboxylic acid type, a sulfonic acid type or a sulfate type. An alkali metal salt of resin acid, a C₈₋₂₀ alkyl sulfonate, an alkylaryl sulfate or a condensate of sodium naphthalenesulfonate with formaldehyde, may, for example, 25 be mentioned. As a specific example of nonionic type, polyvinyl alcohol or its copolymer (for example, copolymer with acrylamide), polyvinyl ether or its

copolymer (for example, a copolymer with maleic acid), polyvinyl pyrrolidone or its copolymer (for example, a copolymer with vinyl acetate), these chemical modified (co)polymer or a cellulose derivative

5 (hydroxyethylcellulose) may be mentioned. As a specific example of cationic type, there is, for example, an aliphatic amine salt or an aliphatic quaternary amine salt. Octadecyltrimethyl ammonium chloride, dodecyltrimethyl ammonium chloride or dilauryldimethyl
10 ammonium chloride may, for example, be mentioned.

However, the polychloroprene latex used for the primer in the present invention preferably comprises the emulsifying agent comprising resin acid and/or an alkali metal salt of resin acid.

15 As the amount of addition of the emulsifying agent and/or the dispersing agent in the polychloroprene latex in the present invention, it is preferably from 0.5 to 20 parts by mass per total 100 parts by mass of an initially charged monomer. If it is less than 0.5 part by mass,
20 the emulsifying power tends to be inadequate. If it exceeds 20 parts by mass, adhesive water resistance tends to deteriorate.

The polymerization temperature of the polychloroprene latex in the present invention is not
25 particularly limited. However, in order to carry out polymerization reaction smoothly, the polymerization temperature is preferably from 0 to 50°C.

As a polymerization initiator, a persulfate such as potassium persulfate, an organic peroxide such as tertiary-butyl hydroperoxide or the like is preferably used. However, it is by no means restricted to them.

5 The chain transfer agent used for polymerization is not particularly limited, and one commonly employed for emulsion polymerization of a chloroprene may be used. For example, a known chain transfer agent, such as a long chain alkyl mercaptan such as n-dodecyl mercaptan or
10 tert-dodecyl mercaptan, a dialkyl xanthogen disulfide such as diisopropyl xanthogen disulfide or diethyl xanthogen disulfide, or iodoform, may be used.

A polymerization terminator (polymerization inhibitor) for the polychloroprene is not particularly
15 limited. 2,6-tert-butyl-4-methylphenol, phenothiazine or hydroxyamine may, for example, be used.

The final conversion of the polychloroprene is not particularly limited and can be optionally adjusted. Unreacted monomer is removed by demonomer operation.
20 However, its method is not particularly limited. With respect to the polychloroprene latex in the present invention, solid content concentration can be adjusted to a required concentration by concentration or dilution by adding water, etc. As a method for concentration, there
25 is concentration under reduced pressure, but it is not particularly limited.

Structure of the polychloroprene latex in the

present invention is not particularly restricted.

However, by adequately selecting and controlling the polymerization temperature, the polymerization initiator, the chain transfer agent, the polymerization terminator, the final conversion of the monomer, demonomer, condition of concentration or the like, it is possible to adjust the solid content concentration, molecular weight of a toluene soluble part, toluene insoluble content (gel content), etc.

10 From the view point of adhesive property, gel content of (co)polymer in the polychloroprene latex is preferably adjusted to from 3 to 60 mass% for balance between initial adhesive strength and normal adhesive strength.

15 The EVA resin of the present invention is a copolymer resin of ethylene/vinyl acetate and is preferably used in an emulsion state. A method to obtain the EVA resin emulsion is not particularly restricted, and a known method can be used. For example, after the
20 EVA polymer is dissolved in an organic solvent, hot water and an emulsifying agent are added and dispersed, whereupon the solvent is removed under reduced pressure to obtain a latex. The organic solvent is not particularly restricted. For example, one which is able
25 to dissolve the EVA polymer such as toluene, xylene, n-hexane or cyclohexane may be mentioned.

As the emulsifying agent, as an anionic type, there

are a carboxylic acid type and a sulfuric ester type. For example, an alkali metal salt of resin acid, an alkyl sulfonate, or a condensate of sodium naphthalenesulfonate with formaldehyde is mentioned. As a nonionic type, there are, for example, a water-soluble polymer type, an ether type, an ester type, an alkyl phenol type and a sorbitan ester type. For example, a polyvinyl alcohol, a polyoxyethylene monostearate, or a sorbitan monooleate may be mentioned.

10 In order to improve the adhesive strength of adherend, the mass ratio of ethylene in the EVA resin in the present invention is required to be at least 40%, more preferably at least 60%.

The solid content concentration of the water base primer of the present invention is not particularly limited. However, it is preferably at most 60 wt%, more preferably from 10 to 30 wt%. If the solid content concentration exceeds 60 wt%, problem that stability of an emulsion system deteriorates, tends to occur, whereby it becomes a practical problem. Further, in either case of a low solid content concentration of less than 10 wt% or a solid content concentration exceeding 30 wt%, it will be difficult to control the thickness of the primer layer within the optimum range after drying. Accordingly, 20 it is preferably within the a range from 10 to 30 wt% from the practical standpoint.

In a case where an adhesive resin is added to the

water base adhesive, its type is not particularly restricted. Specifically, a rosin resin, a polymerized rosin resin, α -pinene resin, β -pinene resin, a terphenol resin, C₅ fraction type petroleum resin, C₉ fraction type petroleum resin, C₅/C₉ fraction type petroleum resin, DCPD type petroleum resin, alkylphenol resin, xylene resin, coumarone resin, or coumarone-indene resin may, for example, be mentioned. In order to obtain enough initial adhesive strength, a resin having a softening point of from 50 to 160°C is preferred.

A method for adding the adhesive resin is not particularly restricted. However, in order to disperse the resin in the primer uniformly, it is preferred to add in the form of water-based emulsion. A method for preparing the water-based emulsion of the adhesive resin is not particularly restricted, and a known method may be used. There is, for example, a method wherein the adhesive resin is dissolved in an organic solvent such as toluene, xylene, n-hexane or cyclohexane, and hot water and an emulsifying agent are added to disperse, followed by removing the solvent under reduced pressure to obtain latex, or a method wherein the adhesive resin is pulverized into particles, followed by emulsion and dispersing. However, the former, which can produce an emulsion of smaller particles, is preferable.

In addition to the above-mentioned, depending on required property, a polyethyleneimine or a

polyethyleneoxide type of a water dispersion polymer, an inorganic compound such as sodium silicate, a metal oxide such as titanium oxide or zinc oxide, a filler such as clay, a curing agent such as isocyanate, a thickener, a film-forming assistant, an UV absorber, an antioxidant, a plasticizer, a vulcanizer, a vulcanizing accelerator, an antifoaming agent, a preservative, etc. may optionally be added to the primer of the present invention.

It is possible to adjust to the viscosity to desired level by adding a thickener to the water base primer. As a specific example of the thickener, polyvinyl alcohol (PVA), sodium polyacrylate, a water soluble polyurethane, an associated polyurethane emulsion, an alkali-swelling type acrylic emulsion, carboxymethyl cellulose (CMC), methyl cellulose (MC), hydroxyethylcellulose (HEC), hydroxypropyl cellulose (HPC), polyvinyl alcohol or a synthetic smectite may be mentioned.

A suitable amount of the thickener is from 0.01 to 10 parts by mass (as calculated as solid content) per 100 parts by mass of the polychloroprene latex. If the amount is within this range, it is possible to prevent cissing of the primer on the surface of an elastomeric material.

The viscosity of the primer may be adjusted to the specification of the coating method and the coating equipment. However, it is preferably from 10 to 15000

[mPa's] (B type viscometer, 25°C, 30 rpm). If the viscosity is less than 10 [mPa's], cissing on an elastomeric material may sometimes be a problem. If the viscosity is higher than 15000 [mPa's], uniform coating
5 may sometimes be difficult.

A coating method and a coating apparatus required for applying the primer of the present invention is not particularly restricted. As a specific example, a spray method, a curtain flow coater method, a bar coater method
10 or a roll coater method may be used. Further, the roll coater method includes rotogravure roll coater method and a reverse rotogravure roll coater method.

However, in a case where coating area is small, a coating method with a brush or a method, wherein the
15 primer is infused in a sponge or a cloth, and a surface of the elastomer material is wiped smoothly, is reliable and preferable.

A coating amount is not particularly limited. However, in order to obtain stronger adhesive structure,
20 it is preferred that a thickness of the primer layer is thinner than a thickness of the adhesive layer. Specifically, from 60 to 110 g/m² (wet) is preferred.

Regardless of the solvent type or the water base type, the primer of the present invention may be applied
25 to various types of adhesives such as a polyurethane type, an acryl type or a vinyl acetate type. Further, with respect to a polychloroprene type adhesive, regardless of

the solvent type and the water base type, the primer of the present invention may particularly preferably be applied.

An organic material to which the primer of the present invention is applied, is not particularly restricted. Specifically, a vulcanized rubber such as butadiene rubber (BR), chloroprene rubber (CR), natural rubber (NR), isoprene rubber (IR), styrene-butadiene rubber (SBR), butyl rubber (IIR), norbornene rubber (NOR), nitrile rubber (NBR), hydrogenated nitrile rubber (HNBR), brominated butyl rubber (BIIR), chlorinated butyl rubber (CIIR), acryl nitrile-isoprene copolymer (NIR) or ethylene-propylene-diene rubber (EPDM), a styrene-butadiene-styrene block copolymer (SBS), a thermoplastic elastomer of e.g. polyester type, urethane type or olefin type, a polyvinyl chloride resin (PVC), an ethylene-vinyl acetate copolymer (EVA), 1,2-polybutadiene (RB), a polyolefin resin or a styrene resin, and an acrylonitrile-butadiene-styrene copolymer (ABS) may, for example, be mentioned.

The porous organic material to which the primer of the present invention is applied is a foam of the above organic material. The foaming method and the material or thickness of the foam are not particularly restricted and may suitably be selected depending upon particular purpose or application and in consideration of the flexibility or strength of the material. As the foaming

method, for example, melt foaming, solid phase foaming or cast foaming may be mentioned. As a specific example of melt foaming, chemical cross-linkage foaming, electron cross-linkage foaming, extrusion foaming, one step
5 pressurizing foaming or two steps pressurizing foaming may be mentioned. As a specific example of the solid phase foaming, a beads method may, for example, be mentioned. As the cast foaming, molding foaming, block foaming (slab foaming), continuous laminate foaming,
10 injection foaming or spray foaming may, for example, be mentioned.

Further, compositions of the foaming agent, cross-linking density, shape, etc. of these materials are not particularly restricted. With respect to applications,
15 these are suitably applied to various fields such as parts of a shoe, a sport apparatus, packaging materials, automobile accessories, building materials, etc.

Now, the present invention will be described with reference to Examples and Comparative Examples, but the
20 present invention is by no means restricted by these Examples. Further, in the following Examples, "parts" and "%" are based on mass unless otherwise specified.

EXPERIMENT 1

Using a reactor having an internal capacity of 3
25 liters, in a nitrogen stream, 100 parts of water, 5 parts of disproportionated resin acid, 0.6 part of sodium hydroxide, 0.7 part of potassium hydroxide, 0.3 part of a

sodium salt of a formaldehyde/naphthalene sulfonic acid condensate and 0.3 part of sodium hydrogen sulfite were charged. After dissolving them, 100 parts of a chloroprene monomer and 0.14 part of n-dodecyl mercaptan were added with stirring. Using potassium persulfate as an initiator, in a nitrogen atmosphere, polymerization was carried out at 10°C. When the polymerization rate reached 90%, an emulsion of phenothiazine was added to terminate the polymerization. An unreacted monomer was removed under reduced pressure to obtain a polychloroprene latex. Further, the polychloroprene latex was concentrated by evaporating water under reduced pressure and adjusted so that its solid content became 60 mass.

EXPERIMENT 2

Using a reactor having an internal capacity of 3 liters, in a nitrogen stream, 120 parts of water, 4 parts of disproportionated resin acid, 0.8 part of sodium hydroxide, 0.5 part of a sodium salt of a formaldehyde/naphthalene sulfonic acid condensate and 0.3 part of sodium hydrogen sulfite were charged. After dissolving them, 100 parts of a chloroprene monomer and 0.25 part of n-dodecyl mercaptan were added with stirring. Using potassium persulfate as an initiator, in a nitrogen atmosphere, polymerization was carried out at 10°C. When the polymerization rate reached 70%, an emulsion of phenothiazine was added to terminate the polymerization.

An unreacted monomer was removed under reduced pressure. Then, pellets of the polychloroprene were obtained by a usual method of a freeze-drying method.

EXAMPLE 1

5 Using the polychloroprene latex obtained in
Experiment 1, to 100 parts of the polychloroprene latex,
5 parts of an EVA resin having a solid content of 32%
(EV-2, manufactured by CHUKYO YUSHI. CO., LTD.) was added
and 50 parts of a terpene phenol resin emulsion (TAMANOL
10 E-100, manufactured by ARAKAWA CHEMICAL INDUSTRIES, LTD.)
was added as an adhesive resin. Further, by adding pure
water, the solid content in the entire blend was adjusted
to 20% to obtain a primer A.

Further, to 100 parts of the polychloroprene latex
15 obtained in Experiment 1, 50 parts of a terpene phenol
resin emulsion having a solid content of 53% (TAMANOL E-
100, manufactured by ARAKAWA CHEMICAL INDUSTRIES, LTD.)
as an adhesive resin, 2.0 part of a sodium polyacrylate-
water based thickener (ARON A-20L, manufactured by
20 TOAGOSEI CO., LTD.), 10 parts of aluminum hydroxide
powder (HIGILITE H-42M, manufactured by SHOWA DENKO K.K.)
as a metal oxide, 2 parts of zinc oxide dispersions
having a solid content of 50%, and a water disperse type
isocyanate compound (Takenate WD-730, manufactured by
25 Mitsui Takeda Chemicals, Inc) as a curing agent, were
added to obtain a water base adhesive B.

An adhesive surface of an EVA resin sponge (foamed

EVA: 2A-1064, manufactured by MITSUFUKU INDUSTRY CO., LTD., size: 20 mm in width x 70 mm in length) as an adherend was subjected to a washing treatment with toluene to remove dust. Then, toluene was removed by drying in an atmosphere of 60% for 30 minutes. 90 g (wet)/m² of the primer A was applied on each surface of two sheets by a brush. Drying was conducted for 5 minutes in an atmosphere of 60°C.

120 g (wet)/m² of the water base adhesive B was applied thereon by a brush, and dried for 5 minutes in an atmosphere of 60°C. Then, both adherends were put together and press-bonded by a hand roller. The following evaluations were carried out with respect to the test specimens obtained by this method.

15 Test for evaluating initial adhesive strength

After 10 minutes from press bonding, the T-type peeling strength was measured at a tensile speed of 200 mm/min by a tensile tester.

Test for evaluating normal adhesive strength

20 After 5 days from press bonding, the T-type peeling strength was measured at a tensile speed of 200 mm/min by a tensile tester.

EXAMPLES 2 AND 3

The test was carried out in the same manner as in Example 1, except that a type of EVA resin was changed as shown in Table 1.

EXAMPLE 4

The test was carried out in the same manner as in Example 1, except that the terpene phenol resin emulsion was not added.

5 100 g of pellets of the polychloroprene obtained in Experiment 2 was added into a reactor having an internal capacity of 1 liter, and 600 g of toluene was added. The mixture was dissolved with stirring. After dissolving, 50 g of methyl methacrylate was added, and
10 graft polymerization was carried out at 85°C for 5 hours by means of benzoyl peroxide as an initiator.

A solvent type adhesive was prepared by adding 2 parts of polyisocyanate (DESMODUR RFE, manufactured by Bayer) into this chloroprene/graft copolymer solution and
15 used in Example 6.

COMPARATIVE EXAMPLE 1

Adhesive test was carried out without using the primer but using a water type adhesive prepared in Example only.

20 COMPARATIVE EXAMPLE 2

The test was carried out in the same manner as in Example 1, except that the primer was prepared without adding the EVA resin.

COMPARATIVE EXAMPLE 3

25 The solvent type primer was prepared by diluting the chloroprene/graft copolymer obtained in Example 5 with toluene to adjust solid content concentration to be 5%.

Test results of the above Examples 1 to 5 and
Comparative Examples 1 to 3 are shown in Table 1.

[Table 1]

	EXAMPLE					COMPARATIVE EXAMPLE		
	1	2	3	4	5	1	2	3
Primer treatment [parts by weight (1)]								
CR latex	100	100	100	100	100		100	
EVA Resem EV-2 (2)	5	-	-	5	5		-	
EVA Resem EV-6 (3)	-	5	-	-	-	No primer	-	Solvent type primer
EVA M-197 (4)	-	-	5	-	-		-	
Adhesive resin emulsion (5)	50	50	50	-	-		50	
Pure water	Proper amount	Proper amount	Proper amount	Proper amount	Proper amount		Proper amount	
Primer solid content [%]	20	20	20	20	20	-	20	5
Type of adhesive	Water base	Water base	Water base	Water base	Solvent type	Water base	Water base	Water base
Adhesive strength [N/mm]								
Initial adhesive strength	4.6	4.5	4.0	4.0	4.5	0.6	1.2	4.5
Normal adhesive strength	5.3	5.1	4.5	4.9	5.3	1.0	2.2	5.1

(1) parts of adding each additive is represented by wet base in a case where polychloroprene latex is 100 parts.
 (2) EVA Resem EV-2: mass ratio of ethylene 72%, solid content concentration 32% (manufactured by CHUKYO YUSHI.CO.,LTD)
 (3) EVA- Resem EV-6: mass ratio of ethylene 81%, solid content concentration 32% (manufactured by CHUKYO YUSHI.CO.,LTD)
 (4) EVA M-197: mass ratio of ethylene 59%, solid content concentration 32%(manufactured by CHUKYO YUSHI.CO.,LTD)
 (5) TAMANOL E-100: terpene phenol type resin emulsion (manufactured by ARAKAWA CHEMICAL INDUSTRIES, LTD.)

[Effects of the Invention]

Obviously as shown in Table 1, the water base primer of the present invention (Examples 1 to 6) has stronger adhesive strength than a solvent type primer, which is a
5 conventional primer, and has high adhesive strength with respect to any of a water type adhesive and a solvent adhesive.

[TYPE OF DOCUMENT] ABSTRACT

[SUMMARY]

[OBJECT]

5 Providing a water base primer which exhibits
excellent adhesive strength by applying it on a surface
of adherend preliminarily before an adhesive is applied,
when bonding adherend such as an organic material or a
porous organic material.

[MEANS OF SOLVING PROBLEMS]

10 By an adhesive method that a water base primer,
characterized by comprising a polychloroprene and an EVA
resin, as essential component is applied on a surface of
adherend preliminarily before an adhesive is applied,
high adhesive strength is obtained.

15 The EVA resin of the present invention is a
copolymer resin with ethylene vinyl acetate, and is
preferably used in the form of emulsion state.

20 In order to obtain higher adhesive strength of an
adherend, mass ratio of ethylene in the EVA resin is
preferably at least 40%, more preferably at least 60%.

[SELECTED FIGURE]

No Selected Figure

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